THE RECOVERY OF

NITRATE FROM CHILEAN CALICHE.

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CONTAINING A VOCABULARY OF TERMS, AN ACCOUNT OF THE SHANKS SYSTEM, WITH A CRITICISM OF ITS FUNDAMENTAL FEATURES, AND A DESCRIPTION OF A NEW PROCESS.

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PREFACE.

SINCE the date of the first manufacture of nitrate from the caliche found in the (then) Peruvian pampa, during the early years of the 19th century, the developments in the industry have been almost entirely in connection with the application of new and improved processes for the purpose of making profitable the treatment of material of decreasing nitrate content. The average ley, or nitrate percentage, of the caliche treated during the first fifty years probably averaged at least 75 per cent. In the most primitive method of beneficiation the rich caliche was boiled with water or mother liquor in large copper pans by the direct application of heat; and the nitrate was then crystallised from the resulting strong solution. The use of steam was adopted in 1850, when the average grade of caliche treated had dropped to about 50 per cent.; but even with this modification the process, if such it could be called, was still hopelessly inefficient, the ripios, or residues discharged, containing from 25 to 30 per cent. of nitrate; and the borra,

or slime, usually more than the original caliche. In 1876, important developments occurred as a result of the introduction by Mr. J. Humberstone —known for many years past as the father of the industry and held in high esteem—of a modification of the well-known Shanks system of progressive enrichment of solutions by lixiviation, as practised in the English alkali industry. The first installation was made at oficina Limeña, for the treatment of rich ripios from earlier processes. The Shanks system was soon adopted throughout Chile, and has to the present day remained the standard method of extracting the nitrate.

The introduction of the Shanks system resulted in notable economies, as compared with previous practice, with the result that caliche of lower ley could be treated at a profit. The grade of the material sent to the maquinas for the 20 years subsequent to the introduction of the Shanks system was probably fixed at a minimum of over 25 per cent., and in most cases averaged well over 30 per cent. nitrate.

With the reduction in grade of caliche being treated, a considerable decrease in percentage extraction of nitrate, by existing methods, was observable. At all times, however, a veil of mystery has been drawn over the actual results of practice, and reliable figures were impossible to obtain.

Accurate estimations of nitrate loss were seldom attempted; neither the caliche nor the various types of ripio discharged could be sampled with accuracy, nor was the dry weight of the several products obtainable. The facilities for the reduction of the samples were—and are to a large extent—such that the resulting analyses could only be considered as approximations. These circumstances permitted a wide latitude in the statement of result, and without fear of contradiction in the case where exceedingly high extractions were reported. Although optimistic figures have been given at various oficinas as to the amount of nitrate extracted, it is improbable that the actual recovery in crystal form and ready for shipment exceeded, on the average, 55 per cent. of the nitrate originally in the caliche; and it is certain that, in very many cases, the amount was considerably below this estimate.

With the exhaustion of the richer deposits of caliche, the general inefficiency of the present process has become more and more apparent, especially in connection with the treatment of material from ground containing any appreciable percentage of borra or slime. Various attempts have been made to improve results, usually by the installation and operation of complicated filtration equipment—ill-suited to the conditions

prevailing—but with no real success, economic or technical, as judged by the failure of the industry to adopt, to any general extent, any of the modifications which have been introduced from time to time.

The Shanks process, thanks to the technical skill and initiative of its sponsor, Mr. Humberstone, has well served its purpose. It has enabled the industry to reach a high state of prosperity; and it has been responsible, in no small measure, for Chile's present-day position in the front rank of progressive nations. Conditions are, however, rapidly changing. Disquieting accounts are current as to the success of synthetic methods for the manufacture of nitrate or nitrogen compounds, perfected by force of circumstances during the Great War; and the beds of caliche remaining untouched must be classed, for the most part, as either too low grade or too "borriente" for successful treatment by existing methods. The present high cost of fuel is likely to persist, or increase; labour is becoming more and more expensive and restive; the present abnormal price of nitrate cannot be maintained indefinitely, and the whole world is in urgent need of cheap fertiliser.

The fact needs emphasis that seldom or never is work discontinued at a salitrera on account of

the absence of caliche, but invariably because of the diminishing amount of nitrate contained, or because of the presence of a prohibitive amount of borra. Enormous dumps of ripio, which are accessible and can be cheaply handled, are scattered over the pampa. Estimates of the amount vary between 100,000,000 and 150,000,000 tons. Although this ripio is untreatable by present methods, and the cost of a filtration process would certainly be prohibitive, it will become a national asset of considerable importance as soon as attention is paid to an alternative process, based on adequate-scale operations, and developed on the foundation of a satisfactory appreciation of the fundamental principles of leaching treatment. The ripio dumps of Chile are also of considerable value as affording a sufficiency of hard, insoluble material with which may be mixed the "borriente" caliche which is now discarded, at some expense, as unsuitable for Shanks-maquina treatment; or only handled at high cost and with indifferent results by one or other of the complicated filtration systems which have been proposed. No estimate is possible of the amount of low-grade caliche in Chile, but it probably amounts to over a billion tons. But this is clearly only available provided that the present small-scale operations and expensive handling methods in vogue are superseded by a

the absence of caliche, but invariably because of the diminishing amount of nitrate contained, or because of the presence of a prohibitive amount of borra. Enormous dumps of ripio, which are accessible and can be cheaply handled, are scattered over the pampa. Estimates of the amount vary between 100,000,000 and 150,000,000 tons. Although this ripio is untreatable by present methods, and the cost of a filtration process would certainly be prohibitive, it will become a national asset of considerable importance as soon as attention is paid to an alternative process, based on adequate-scale operations, and developed on the foundation of a satisfactory appreciation of the fundamental principles of leaching treatment. The ripio dumps of Chile are also of considerable value as affording a sufficiency of hard, insoluble material with which may be mixed the "borriente" caliche which is now discarded, at some expense, as unsuitable for Shanks-maquina treatment; or only handled at high cost and with indifferent results by one or other of the complicated filtration systems which have been proposed. No estimate is possible of the amount of low-grade caliche in Chile, but it probably amounts to over a billion tons. But this is clearly only available provided that the present small-scale operations and expensive handling methods in vogue are superseded by a

process such as is suggested in the following pages.

Many industries have passed through a similar stage of evolution, and the history of metallurgical progress is replete with examples of the gradual abandonment of mineral deposits and residue dumps, because of lowness of grade or refractoriness, followed in due course by improvements in practice and the successful establishment of alternative processes on an adequate scale. An English mining engineer, with an intimate knowledge of affairs in Chile, published a book in London in 1826 in which he gave conclusive "proofs" of the "fallacious notions" entertained in certain quarters as to the possible productiveness of Chilean copper mines. Yet in the sixties, Chile was producing nearly 70 per cent. of the world's output of the red metal, and to-day she possesses in successful operation what is perhaps the largest copper mine in the world, in addition to a number of smaller, though exceedingly valuable, properties. All this has resulted from technological advance and large-scale operations.

The same degree of development and ultimate success is possible in the nitrate industry, even though the most sanguine estimates in connection with synthetic nitrogen products be realised, provided that a bolder policy is adopted and less

attention paid to the application of patented machines and more to the fundamental principles of simple leaching, operated on a scale commensurate with the size and grade of the remaining deposits.

The purpose of this booklet is to point a way to a more economical and efficient process which preserves the simplicity of the Shanks system and, at the same time, permits of the complete treatment, to a satisfactory and logical conclusion, of material which is now considered valueless. No estimate can be made at the present time as to the ultimate benefit to the industry by the adoption of the system described—the most conservative estimates are staggering; but it appears certain that the enormous increase permissible in any estimate of the available reserves of raw and treated material, coupled with a radical decrease in the cost of treatment should, in the near future, materially aid in the reduction of the present high cost of living throughout the world.

A. W. ALLEN.

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VOCABULARY OF TERMS
USED IN CHILEAN NITRATE TECHNOLOGY.

Agua feble. The solution remaining after the refrigeration of **agua vieja** or other nitrate liquor.

Agua vieja. A term used to refer to the mother liquor, at atmospheric or below atmospheric temperature, remaining after the fractional crystallisation of commercial nitrate from **caldo.**

Batea. Crystallising vat.

Borra. A term used indiscriminately to refer to (1) the mixture of slime, crystallised nitrate, sand, water, or other impurities which passes through or is found underneath the perforated plates supporting the **caliche** in the boiling tank; or (2) the exceedingly fine earthy material associated with or forming part of the **caliche.**

Cachucho. The boiling tank in which the high-grade nitrate liquor is made by means of evaporative concentration, and where the remaining nitrate associated with the **caliche** is displaced by weak solutions and water.

Caldillo. A somewhat indefinite term referring to any solution which does not approach a **caldo** in nitrate strength, although containing more nitrate than a mother liquor.

Caldo. The liquor of the highest density and temperature. The first liquor drawn from the **cachucho.**

Caliche. Nitrate bearing material containing, in addition to sodium and potassium nitrates, soluble impurities in a number of forms and in widely varying proportions.

Cateo. The survey of a **salitrera,** for the purpose of forming an estimate of tonnage and nitrate content of **caliche.**

Chullador. An open, flat-bottomed vat, used for the clarification of **caldo** by the settlement of entrained **borra** and salt.

Crinolina. The perforated-plate false bottom on which the charge of **caliche** rests in the **cachucho.**

Cuaja. The deposit of crystals in the **batea,** or crystallising vat. The term is usually used to refer to the size of the deposit or the quantity of nitrate produced.

Ley. A term used to represent the richness, or the percentage of nitrate in **caliche** or other material.

Maquina. **Caliche** treatment plant.

Oficina. A nitrate property equipped with **maquina.**

Relave. Weak nitrate liquor, added before the final water wash to displace the higher-grade liquors and to dissolve residual nitrate.

Ripio. The residue, or waste product.

Salitre. The commercial nitrate product.

Salitrera. Nitrate bearing ground.

RECOVERY OF NITRATE FROM CALICHE.

THE SHANKS PROCESS.

The Shanks process, in use throughout Chile for the recovery of nitrate from caliche, consists, essentially, of a reduction of the raw material in rock-breakers to about 3-inch size. This is then carried by belt conveyors or gable cars to boiling vats. In a few instances secondary crushing is practised, but in the majority of cases the caliche passes direct from a small storage, through the rock-breakers, and direct to the vats.

The vats, or cachuchos as they are called, are rectangular in shape, and are divided horizontally by a crinolina, or perforated-plate false bottom placed about 9 inches above the floor of the vat, into (*a*) the upper portion containing the caliche and steam coils, and (*b*) the lower portion connected with solution exit pipes and doors for the discharge of the ripio.

A system of solution transfer from cachucho to cachucho, with exit at or from the bottom and inlet at the side of the vat, provides for the pro-

gressive enrichment of solution by an imperfect displacement movement. In the vats recently charged with caliche the solution is boiled or kept at a high temperature and water evaporated, so that a high-grade caldo may be produced for crystallisation. This is withdrawn from the space underneath the crinolina and replaced at the same time with solution from the charge from which caldo had been previously drawn. The caldo from the cachuchos usually contains borra and has to be clarified before it can be sent to the bateas. This operation is carried out in a chullador, the solution being mixed with various settling agents such as flour in the form of a thin paste, and guano, and decanted as soon as the desired result is obtained

The solution from the chullador is laundered to one of a number of bateas, where the nitrate is allowed to crystallise. The residual mother liquor, the agua vieja, is returned to the storage tanks, from which it is drawn when required and sent to the cachuchos, thus completing the cycle of operations.

An excessive amount of hand-picking at the salitreras, to minimise trouble with borra in subsequent treatment, results in high mining expense. The cachuchos are small and fitted with steam pipes, and must, therefore, be discharged by hand. Special wages have to be paid for disagreeable work, and only experienced men are of use. Some of the ripio is discharged in a sloppy condition, thus making cheap residue disposal difficult.

After the completion of treatment, an exceedingly high-grade mixture of sand and borra is found underneath the crinolina. This, in some plants, is re-treated by special classifying, decantation, and filtration apparatus, and the nitrate content reduced to an amount which usually varies from 4 to 12 per cent. In some instances the fines associated with the dry crushed material are separated from the coarse. These fines may be treated alone or together with the borra and sand found underneath the crinolina.

The accurate accounting of results is an impossibility. As many as four types of ripio may be discharged to the dumps, the nitrate in the proportion coming from above the crinolina plates in the cachucho being invariably used as an index to the efficiency of operations in the maquina. Between theoretical extraction and actual recovery there is seldom an attempt at agreement. Because of the difficulty of sampling so unhomogeneous a mass as the material fed to the cachuchos, the impossibility of estimating the tonnages of the various ripios discharged, and the wide variation in nitrate content in the individual pieces comprising the ripio from the cachuchos, the actual yield of nitrate usually indicates a loss amounting to twice or even three times as much as the estimate of nitrate in ripio would seem to indicate.

Sampling of the caliche in the first instance, and before it goes to the cachuchos, is usually an inefficient operation, resulting in a faulty estimate. In many instances the fines carry a higher per-

centage of nitrate than the coarse. The personal equation, therefore, governs the estimated ley of the material sent to the cachuchos. Shovel sampling usually results in the inclusion of an abnormal proportion of coarse lumps, and the lower amount of nitrate recorded tends to counterbalance some of the unrecorded losses occurring during treatment. The early exhaustion of normal-ley caliche from the salitreras is usually attributed to a faulty cateo in the first instance. Certain parts of the pampa were assumed to have caliche of a certain ley; this was not realised when the material was sent to the maquina. These and similar explanations are usually accepted, in spite of the fact that extensive cateos on a proper basis have invariably indicated that discrepancies occur which influence over, as well as under, estimation and to an extent that leads to the maintenance of the original average.

SHANKS PROCESS—THE EXTRACTION STAGE.

An exceedingly unhomogeneous product, usually direct from swing-jaw breakers, is sent for treatment. The breaker jaws wear at a maximum in the centre; and a small or large percentage of oversize, depending on the age of the jaw plates, passes to the cachucho. The feed, therefore, consists of large lumps, the ordinary proportion of medium sizes, and a small amount of fine dust or borra. With a mixture containing such widely varying sizes, the average length of treatment cannot be regulated to ensure efficiency of result as well as economy of time.

The caliche is delivered to the cachucho either by means of gable cars or belt conveyors delivering to one spot at a time. No system of distribution, to prevent segregation, is attempted. The fines are piled at several points in the vat, and the coarse lumps find their way to the edges. Only one advantage is apparent from the segregation resulting: the maximum amount of solution passes around the larger lumps, which are, therefore, given a proportionately greater change of solution than the finer material, in which the nitrate is more available. In many cases, however, the fines escape adequate treatment, the ripio taken from

various parts of the cachucho often differing in nitrate content from 2 to 10 per cent.

Enrichment of the Charge. — The inefficiency of nitrate recovery—the lengthy time of treatment to obtain even moderately low ripios—is largely due to the enrichment of the charge by the evaporative concentration of high-grade solutions and by the addition of caldillo and agua vieja. These high-grade nitrate solutions have low dissolving powers. Caldo is formed mainly by evaporative concentration, and not by the abstraction of nitrate from the caliche; and this involves the augmenting of the nitrate contents of the cachucho, resulting in a proportionately high ripio. The boiling or high-temperature maintenance disturbs the equilibrium of the charge, causing the borra to circulate with the solution and finally to find its way underneath the crinolina, and from thence to the surface of other charges, or to the relave tanks.

Displacement Stage. — Caldo when formed is largely diluted with incoming caldillo. This is due to the inefficiency of the displacement method adopted and the short circuiting of the solution through the charge.

As much caldo as possible is drawn from underneath the crinolina, or perforated-plate bottom supporting the charge, and partly displaced by the caldillo from the previous charge; and delivered, not on to the top of the caliche, but into the side of the charge, and generally at the opposite end of the cachucho.

Efficient displacement of solutions during leaching must take place in either an upward or downward direction. This involves either (*a*) pumping from vat to vat in any system of solution concentration; or (*b*) the erection of the vats at different levels. The latter practice would present obvious disadvantages. In the Shanks system an effort is made to secure displacement from vat to vat without recourse to either pumping or variation in vat level. The result is that operations are considerably simplified, but at the expense of efficient displacement.

Lengthy treatment and large amounts of water are needed, not only to displace the high-grade solution left in contact with the caliche after the concentration stage, but to effect dissolution of the nitrate left untouched on account of the low dissolving powers of the solutions used in the earlier stages of treatment. The inefficiency of the work done in the displacement stage is, however, largely due to the fact that, in the Shanks process, no true displacement of solution takes place other than when circulating in individual cachuchos; and any good effects in the latter case are nullified by the circumstance that nitrate is removed by the solution only to be again put in contact with the charge. So that, whereas the undissolved nitrate in the charge is lessened in amount, the dissolved nitrate in the liquid surrounding the charge, a proportion of which must necessarily be left in contact with the caliche as moisture or entrained liquor, is higher in nitrate

than before that phase of treatment was commenced. It may be taken as an axiom that, in all efficient leaching operations, the concentration of the solution must be accompanied by progressive impoverishment of both the charge and the solution in contact with it.

Five weak features are, therefore, apparent in the extraction stage of the process :

1. Unsuitable and unhomogeneous character of the caliche when prepared for treatment.
2. Lack of adequate support for, or facilities for drainage from, the charge.
3. High nitrate content of material in the cachuchos after the concentration stage and before the displacement stage, leading to correspondingly high ripios, and due to the practice of forming caldos in contact with the caliche.
4. Imperfect displacement of dissolved nitrate in solution by the present horizontal-delivery system of solution from cachucho to cachucho.
5. Disturbance of the distribution of borra throughout the charge by boiling or high-temperature maintenance, with contamination of solutions and loss of nitrate.

Evaporation of Water in the Cachuchos.—A necessary step in the concentration of liquors to caldo strength is the evaporation of water; and this is effected to a large extent by boiling or high-temperature maintenance in the cachuchos. An

amount of water, usually equivalent to from 25 to 30 per cent. of the original weight of caliche, is added during treatment for displacement and dissolving purposes. About 50 per cent. of this is discharged with the ripio as moisture. The remainder must be evaporated. Vaporisation is effected concurrently with the concentration of liquor in the first stage of treatment. A large expenditure of heat is necessary. Radiation losses are high, both from the bottom and sides of the cachuchos, and arising from the chilling of the surface of the charge with cold air. The intermittent heating—and exposure to low atmospheric temperatures—of large areas of metal for small tonnages handled is also the cause of considerable heat loss.

Action in the Chullador.—Caldo drawn from underneath the crinolina, as described, must be clarified before going to crystallisation. It is claimed that the action in the chullador is also one of salt crystallisation; and this is explained by the assumption that, with increasing concentration and rise in temperature in the caldo-forming stage, dissolving power is available, which is used up in dissolving salt which had been precipitated during the early stage of concentration or is present in excess in the caliche, to an amount above that which would have been taken up had both nitrate and salt been available in equal or equivalent proportions. In other words, salt was available in excess during the first stage of concentration, but nitrate, though present, was not available, being

occluded in the larger lumps of caliche. This resulted in a gain in concentration of salt during the first drop of temperature in the chullador. The explanation is plausible, but it is also evident that some, if not most, of the salt deposited during "chullaring" enters the chullador in the form of a crystal suspension.

Whatever the exact function of "chullaring," it is evident that its necessity arises from:

1. Muddy solutions.
2. Salt in suspension, or lack of available nitrate in the charge, or both.

Muddy solutions are caused (*a*) by the disturbance of the equilibrium of the charge by boiling or high-temperature maintenance in the cachuchos, and the freeing of an unnecessary amount of borra; (*b*) uneven resistance to flow of solution through the charge, resulting in the short-circuiting of solutions; and (*c*) the incomplete and unsatisfactory support for the charge by means of punched plates, leading to local collapse at numerous small areas, and the washing through of borra and sand.

Lack of available nitrate is the result of insufficient exposure of the nitrate in the caliche to the solvent action of the solution. The caliche is crushed insufficiently fine.

The operation in the chullador consumes time, involves loss of heat which should be utilised to effect evaporation of water, necessitates the addition of an expensive settling agent such as flour, and lowers the efficiency of extraction in the

cachuchos by the necessity of increasing the nitrate content of weak solutions which must be used to wash the salt and entrained nitrate left in the chulladores after the decantation of caldo. The necessity for "chullaring" prevents the attainment of a maximum degree of nitrate concentration in the caldo, thus excluding salt.

Modifications in Operation in the Extraction Stage of Treatment.—Apart from logical improvements in the matter of (*a*) the substitution of efficient evaporation for that proportion of the necessary evaporation over and above what is required to concentrate solutions to the required strength in the cachuchos; (*b*) the more accurate estimation of caliche tonnage; and (*c*) the reduction in nitrate content of agua vieja by refrigeration, the modifications in treatment introduced during recent years have been almost entirely in connection with the possible application of certain patented machines for the special treatment of the borra, or a sand-slime product that may be obtained by the classification of the crushed caliche. Complications have been introduced, in most cases without obvious benefit; and, without going into detail as to the various attempts made along such lines, it may be said that:

1. There is no justification for the extra expense involved in the classification of the small amount of "borriente" material found in normal crushed caliche, and the treatment of the fines by any system of pressure or vacuum filtration.

2. Owing to the refractory nature of the borra after the absorption of the nitrate solution, and the difficulty, if not impossibility, of thickening or filtering a borra pulp by any known method, it is highly inadvisable to separate this from the sandy material, either purposely or as an unavoidable phase of any scheme of treatment adopted.

No convincing or consistent data have been available to date as a result of the introduction of any filtration process to handle the whole or part of the product, neither have consistently low ripios been secured, even after the expenditure of considerable sums of money for the purchase and installation of complicated equipment, usually involving ample power provision. If it is proposed to treat the whole of the caliche by pressure or vacuum filtration, then it is evident that the expense would be prohibitive ; and that the nitrate produced from such a process, as far as normal and low-grade caliches are concerned, could not ultimately compete with synthetic nitrate produced by improved methods. If only a part of the caliche is to be treated, then the efficiency of nitrate extraction will decrease, with certain variations, as the proportion of borra increases.

The complete, or nearly complete, recovery of nitrate from sandy material is, of course, entirely feasible and practicable on a commercial scale by the adoption of one of a number of methods of continuous or intermittent operation, such as may be seen in every-day practice in other industries.

The extraction of nitrate from borra is an entirely different proposition, because of the fact that low ripios can only be secured, after the particles have been allowed to absorb high-grade nitrate solution, by a slow process of diffusion, and depending for its efficiency on time available and the difference in density of the replacing solution as compared with the density of the solution in the borra particles. High pressure or vacuum applied to the replacing solutions, in order to insure passage through a compact mass of borra, are both valueless as far as the impoverishment of the solution held by the particles is concerned. If nitrate absorbed in this manner is to be recovered, then the borra particles must be kept surrounded with the solution of gradually decreasing strength for a considerable time, to allow for the equalisation of pressures. Without inordinate expense for equipment and operation, it would be impracticable to give the necessary time for satisfactory displacement in any filtration system. With efficient gravity leaching it is invariably found that the colloid slime associated with the residue is lower in dissolvable salts than would be the case if it were isolated and given a normal treatment involving pressure or vacuum. As far as borra is concerned, although the underlying principles are theoretically attractive, it would appear that any system of countercurrent decantation is out of the question.

SHANKS PROCESS—THE CRYSTALLISATION STAGE.

The caldo from the chulladores is delivered by launder to one of a number of bateas, or crystallising vats. A "field" of these necessarily covers a large area, so that cooling of solution *en route* to the more distant bateas is inevitable, and considerable crystallisation of nitrate usually takes place in the launders. A proportion of the fine crystals formed passes along with the caldo, and is deposited in a heap in the batea directly below the point of delivery from the launder. The remainder is removed by hand labour. The loss of heat during "chullaring" and transfer of caldo to the bateas is shown by the fact that with an initial temperature of 105° C. in the liquor flowing from the cachucho the temperature of the solution in the batea, when full, might be 60° C., or even less.

Initial cooling in the batea is exceedingly rapid, and is the result of radiation from the sides and bottom, all of which are exposed to low night temperatures. Evaporation is restricted and radiation increased by the formation of a scum on the surface of the liquor, for the breaking up of which no provision exists other than by means of hand labour.

The caldo is left in the bateas until a sufficiently low temperature is reached or a certain deposit of nitrate has taken place—a tedious process, usually involving from 7 to 10 days' exposure. Temperature drop is exceedingly irregular, owing to the viscosity of the liquid and the acquisition of heat derived from the sun during the daytime. Under present circumstances the semi-cooled caldo is re-heated daily in the sun, evaporation is slight on account of average low temperature, cooling is retarded, and large batea space is required to produce a small amount of atmospheric-temperature agua vieja.

Loss of heat is an essential concomitant to, and an indication of, the degree of evaporation under efficient conditions. If evaporation takes place at a low temperature, then the heat required to vaporise unit quantity of water is considerably more than is required to vaporise the same quantity of water from liquor at a higher temperature. With batea operation most of the heat available for evaporation at the higher temperatures is dissipated by conduction losses and direct contact with excess cold air. When the solution has reached such a temperature that evaporation is slight, then the deposit of crystals on the sides and bottom of the batea prevents or delays the chilling of the liquor to low temperatures. Excessively large batea installations are, therefore, necessary to counteract the inefficiency of operations at every stage of the process.

Reduction in solution density during batea

crystallisation is retarded by the supersaturated condition of the liquor, which indicates that temperature decrease is only one of several factors affecting the deposition of nitrate. The maintenance of this condition of supersaturation is helped by the quiescence of the liquid, a fact that is evident when agitation is practised, and an immediate crop of crystals is produced.

The initial formation of the crystal is an important phase of operations of this kind. In batea practice and after the first few days of exposure this can only come about by surface chilling, caused either by evaporation or by heat conduction to passing air currents. This cooling action is retarded in the day-time by the transference of heat from the sun to the surface layer of the liquid in the batea, thus effectively insulating the remainder from maximum action at night. Surface crystal formation is delayed, the movement of the liquid is practically nil for several days, and a condition of supersaturation and stagnancy is maintained which delays operations and necessitates extensive equipment.

The unsatisfactory features of this phase of the process may be summarised as follows:

1. Loss of heat, and crystallisation in launders, formation of small crystals from agitation of liquid in transfer, both due to single-plane batea system and intermittent operation.
2. Loss of heat from liquor sent to batea, without appreciable compensation.

3. Enormous batea capacity, due to (*a*) inefficient and slow cooling, and (*b*) time lost for drainage and in hand discharging.

No notable changes have been made in the standard practice of crystallising nitrate from caldo other than in connection with the production of high-potash nitrate from the refrigeration or fractional crystallisation of agua vieja or intermediate liquors, and the production of agua feble. This has resulted, incidentally, in an increase in the yield of nitrate from caldo and a reduction in quantity and grade of the liquor returned to the cachuchos.

GENERAL RESULTS FROM PRESENT METHOD OF TREATMENT.

With average output, normal time of treatment, and by using reasonable amounts of steam and water, the principal defects of the Shanks process are seen in:

1. High loss of nitrate in ripio and borra, restricting the grade of caliche that can be treated at a profit.
2. High cost of operation, arising largely from the inefficient manner of evaporating water, high radiation losses, and small unitary tonnages.
3. Inefficient accounting of results, due to the lack of provision or inability to sample

with accuracy the incoming caliche or the outgoing ripio and borra.

4. Small units, necessitating large areas of equipment, restricting the capacity of the maquina, and making impossible the reduction of overhead expense to reasonable limits.
5. Comparatively high labour requirements, owing to absence of, or impracticability of installing, mechanical provision for the handling of caliche at the salitreras and ripio and nitrate at the plant.

FEATURES OF DESIRABILITY IN ANY ALTERNATIVE SCHEME OF TREATMEMT.

A desirable process, to justify consideration, must indicate the prospect of radical reduction in labour requirements, operating cost, and nitrate content of ripio: it must appear a feasible method for the ultimate re-treatment of the ripio dumps in Chile, and it must be available as a basis for convincing argument to use against the contention that any system of synthetic nitrate production during the life of the Chilean deposits is commercially feasible during times of peace, however much the establishment of plants for this purpose may be desirable from the military point of view.

Any alternative scheme of treatment should, preferably, be adaptable, for small-scale operations, to existing equipment without radical or expensive alteration, and without sacrificing too many of its basic advantages. In summary, the aim should be to evolve a process to ensure:

1. Minimum nitrate loss in ripio, as well as during treatment.
2. A general cost reduction, both with regard to first cost of plant, amortisation, and operating expense.
3. A general labour reduction, by the substi-

tution of modern mechanical methods for the handling of caliche and ripio.

4. The retention of all the favourable features of the present method: (*a*) simplicity of operation, (*b*) low depreciation, and (*c*) high grade of product.
5. The practicability of the accurate accounting of nitrate losses and recoveries, thus placing the industry on a plane with other highly specialised technical operations.

FUNDAMENTALS OF ALLEN EXTRACTION PROCESS.

The new extraction process (patent applied for) consists essentially of passing a solution or other solvent through a homogeneous mass of caliche which has been dry-crushed to such a size that no appreciable movement of the borra takes place in the direction of percolation. The outgoing solution, therefore, contains only a minute amount of borra in suspension. The charge must be of even and homogeneous texture and free from inequalities as to nitrate content; and in order to achieve efficient results the degree of crushing must be such that the interstitial spaces between the particles are small and evenly distributed. On the other hand, regular though slow percolation is desired, so that crushing must not be carried to a point which would result in the production of so great a quantity of fines that solution passage would be retarded below the economic minimum. On certain classes of caliche it has been found that a reduction so that the whole product passes a $\frac{1}{3}$ or $\frac{1}{2}$-inch square-opening screen results in a desirable mixture through which percolation is possible at a rate permitting the rapid leaching of the soluble salts. In other cases, however, coarser crushing might be more economical or finer crushing more desirable. The

rate of dissolution of the nitrate, other things being equal, will depend on the fineness to which the caliche has been crushed, and also on the rate of percolation of the dissolving solution. Excessive comminution generally results in impermeability of the charge, so that there is, obviously, a limit to which the degree of crushing should be carried.

Essential Apparatus.—The vat to be used for leaching purposes may be of any convenient or desirable size or shape. It is fitted with a bottom support for the charge, of sand, coco matting, or other suitable material which will permit the passage of the solution. This bottom is fixed at an appropriate distance from the floor of the vat, usually about 8 inches; and the space between the two is connected with the necessary inlet and outlet solution, steam or water pipes. The bottom support for the charge may be provided with shovelling slats of wood, or rails; or other means may be taken to prevent damage to or removal of the top layer of the support during the discharge of ripio. This may be effected either through bottom or side doors, or, perferably, by means of mechanical excavating apparatus.

The permeable bottom support in no way acts as a filter. If that were its function it would choke almost immediately after downward leaching was commenced. Its purpose is to offer adequate support for the charge, preventing any movement whatsoever of solids in the direction of the bottom space; it allows for the even distribution of solution during upward percolation, thus obviating

channeling and movement of the borra, and it permits efficient drainage of the charge at the conclusion of leaching, even when a high percentage of colloid slime is present, by permitting the application of a vacuum.

Operation.—One of the features of the process is that the fines are so intimately mixed with the coarser material that, given an even distribution of dissolving solution, there is no tendency for the individual particles of borra in the charge to become segregated or to form an appreciable hindrance to the passage of the solution. Only a minute percentage of the borra in the vat is in contact with or is able to reach the bottom support, or is on the surface or able to reach the surface of the charge. A clear liquor is, therefore, obtained, and whether upward or downward percolation is being practised. In the latter case there is absolutely no clogging of the material used for the support of the charge.

A great advantage in the new process is the ability to percolate the solution in an upward direction. The denser liquor resulting is, therefore, delivered from the surface of the charge, thus obviating crystallisation troubles in piping or other enclosed parts of the apparatus; and the heat in the dissolving solution is conserved by direct delivery underneath the bottom support.

Contact of caliche with solution in the first instance is preferably effected by upward submergence—*i.e.*, the solution, usually as hot as possible, is delivered into the space between the

bottom support and the floor of the vat; and is then, by gravity or other pressure, allowed to saturate the caliche and finally submerge it. An allowance must be made for the difference in density of the inflowing and effluent solutions. This may be counterbalanced by providing extra head for the former.

The leaching of the charge may be continued by upward percolation, the effluent solution overflowing from one vat either going to the crystallisation department or being re-heated and delivered underneath the bottom support in another vat, and so on; the final operation being, of course, gravity drainage with or without the assistance of a vacuum. Or the direction of flow may be changed, and the solution may be drained from the bottom of the charge and replaced, at the same time, by the same or other solution applied at the top. This latter method, however, has obvious disadvantages, especially during the earlier stages of treatment.

The percolation of the charge may be continuous or intermittent, but owing to the comparatively small amount of excess solution in contact with the caliche at any time it is possible to displace one solution with another, or with water or other wash, without appreciable admixture of the two. It is, therefore, usually found preferable to work the process so that a continuous flow of solution passes through the charge; but intermittent leaching may be practised, if found desirable: the charge at certain stages of treatment may be wholly or partly drained of the excess solution

in contact with it, and before the application of the next wash or solvent.

The first high-grade solutions resulting are abstracted from the system and sent to the precipitation or crystallisation department. The weaker solutions are preferably re-heated to insure additional dissolving power, and delivered to the other vats in the series, as practised in ordinary leaching operations in many industries. The solution resulting from the final water wash goes forward in the system as a weak relave.

Advantages of the Process.

1. Even percolation, because of the homogeneity of the charge.
2. Improved extraction of nitrate, because of the comparative fineness of the material.
3. Shorter time of treatment on account of the availability of the nitrate and the efficient displacement of solutions.
4. Quick dissolution of the nitrate in the caliche on account of the continuous displacement of dissolving solutions.
5. Ability to extract nitrate from caliche or Shanks-maquina ripio with the avoidance of concentration of solution into caldo in contact with the charge.
6. Full opportunity for the accurate accounting of nitrate losses and recoveries, thus removing one of the most serious bars to technical progress in the Chilean nitrate industry at the present time.

7. Efficient recovery of the nitrate from the borra without the expense involved by its isolation and treatment as a separate product, together with the elimination of troubles resulting from the contamination of solutions with slime.
8. Retention of all the favourable features of the present Shanks process, with increased yield at a considerably reduced operating cost, especially if utilised on a scale commensurate with the size of the low-grade deposits and ripio dumps in Chile.

The process differs from the Shanks system in that:

1. The caliche is crushed much finer.
2. Percolation and solution displacement are either in an upward or downward direction throughout treatment.
3. Adequate bottom support for the charge is provided which, being permeable, permits passage of the solution without movement of the borra.
4. Heat for the dissolution of the nitrate is supplied, in whole or part, by raising the temperature of the solution before it enters the vat.
5. The space containing the caliche in the leaching vats is devoid of steam coils, and no boiling or heating of solutions from an external source occurs when the liquor is in contact with the charge.

6. Particular care is taken with regard to the thorough mixing (by delivery into bins of ample storage capacity) of the various classes of caliche when crushed, as well as the even distribution of the mixture in the vat.
7. Effective insulation of vat and solution contents is feasible by ordinary means, particularly on account of the ability to cover the solution on the charge with wood, float insulators, and the practicability of bucket grabbing the ripio, thus obviating the need for discharge doors and tracks underneath the vat.
8. No appreciable evaporation takes place in the vat. Caldo can be produced by one of a number of methods, such as the re-heating and re-circulation of effluent solutions ; but, in view of the importance of low ripios and rapid treatment, emphasis is laid on the practicability of concentrating solutions by efficient means outside the vat.
9. Concentration of the solution in the leaching vat is a non-evaporative process ; and the enrichment of the percolating solution is accompanied by the impoverishment of the caliche. Solution displacement is continuous, so that the impoverishment of the caliche is accompanied by a corresponding reduction in the grade of the solution surrounding the charge—the ideal conditions for efficient extraction of solubles.

HEATING OF NITRATE SOLUTIONS.

Two important considerations must be borne in mind in connection with the heating of nitrate solutions:

1. Evaporation must be minimised, to avoid the deposition of salt and other impurities.
2. Effective measures must be taken to prevent stagnancy of solution in contact with the heating surface, resulting in local high temperatures, inefficient heat transfer, and accretions.

Assuming the use of iron or steel steam pipes as the most convenient means of heat supply, the efficiency of transference will depend to a great extent on the velocity of flow of the steam and the degree of movement of the liquid in contact with the pipes. By adopting a system of counter-current flow of steam and liquid it is possible to secure highly efficient operation. Evaporation arises almost entirely from the exposure of high-temperature solution to the atmosphere, so that counter-current heat transfer is additionally desirable, because of the fact that high temperatures are only reached when fresh steam is encountered —*i.e.*, when the solution is ready for discharge.

A special type of heater (patent applied for) has

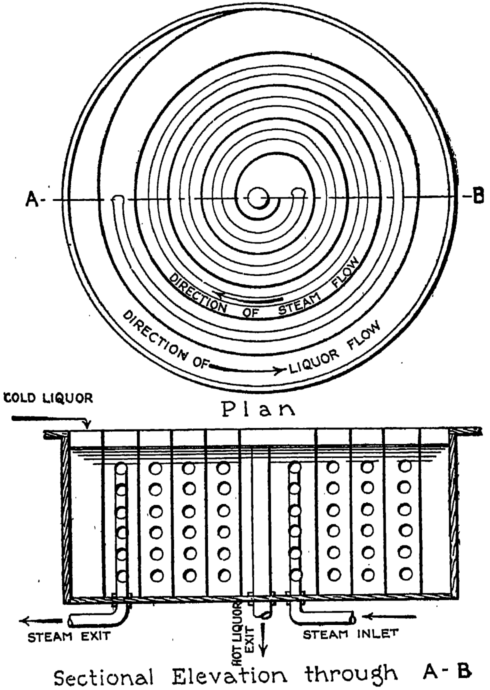

Fig. 1.—Diagram showing Design of Solution Heater.

been developed, a diagram of which is shown in Fig. 1, which has proved thoroughly satisfactory in practice. It will be seen that:

1. Radiation losses are reduced to a minimum.
2. Effective movement of both steam and solution is ensured.
3. There is practically no evaporation of water.
4. The apparatus is simple in design and the parts are accessible.

ACTUAL OPERATING RESULTS FROM NEW PROCESS.

The fundamentals of the new process have been amply demonstrated on both a small and large scale, and with entirely satisfactory results. Practically every type of caliche met with in Chile has been treated. In view of the trouble experienced with "borriente" material in ordinary Shanks-maquina operation, tests have been made on caliche mixtures containing an abnormal percentage of borra. The new process is essentially a leaching process and not a filtration one—a limited pressure is available to assist the passage of solution through the mass—but it has been demonstrated by large-scale operation that the amount of borra that can be successfully treated (with over 90 per cent. extraction of the nitrate) is considerably above the amount contained in an average mixture of caliches from the most refractory pampas in Chile. It is now obvious that the expense involved in the hand-picking and sorting of caliches on many pampas is entirely unnecessary. A much lower-grade mixture can be treated at a profit by the new process, and containing an amount of borra which would be deemed prohibitive so far as Shanks-maquina operations are concerned.

In summary, actual operations have shown :

1. Clear solutions at all times.
2. Rapid treatment, usually averaging from 24 to 48 hours.
3. Greater ease in the discharging of ripio, on account of the absence of steam pipes in the vats.
4. Ability to apply vacuum before discharge, thus reducing average moisture percentage in ripio.
5. Excellent results in the treatment of ordinary Shanks-maquina ripio, showing extractions of over 90 per cent. of the nitrate in 24 hours.
6. Ability to treat successfully through a depth of caliche in considerable excess of Shanks-maquina limits, and in large vats.
7. Low total nitrate loss in treating ordinary caliche mixtures, showing an actual extraction of nitrate which is usually between 90 and 95 per cent.
8. Ability to sample caliche and ripio with accuracy and by ordinary means, thus ensuring the correct estimation of nitrate losses and recoveries and the elimination of uncertainty as to result.

THE PRODUCTION OF COMMERCIAL NITRATE FROM LIQUORS EXTRACTED BY THE NEW PROCESS.

If it is thought desirable to sacrifice the advantages arising from the adoption of a system whereby concentration of the solution in contact with the charge is restricted, in favour of what may be considered a feature of simplicity of Shanks-maquina operations, caldo production in the leaching vat can be secured by a number of methods. These include: (1) the re-heating and re-circulating of effluent solutions; (2) the heating of the caliche in the vat by means of high-pressure steam, low-pressure hot air, or flue gases; and (3) the heating of the caliche before delivery to the vat.

Although caldo production in the leaching vat presents favourable features, there are advantages to be gained in the adoption of a process whereby a lower-grade solution is formed in contact with the charge. By the precipitation and re-dissolution of nitrate or by evaporative concentration, or both, a caldo may then be produced as a separate phase of treatment.

Precipitation of Nitrate from Caldillo, followed by Re-dissolution of Crystals to form Caldo.— The success of the majority of leaching processes depends on the feasibility of the con-

tinuous passage of a solvent solution through the charge; and, so long as a certain minimum concentration is exceeded, the deposition outside the vat, by a precipitation process, of the soluble material so extracted. In the leaching treatment of caliche the crystals deposited by chilling from comparatively low-temperature solutions would contain salt. Further, the chilling of a large quantity of weak caldillo would present obvious disadvantages.

The basic idea of precipitation of dissolved material and return of impoverished solution for extraction purposes would be feasible in the instance under review by (*a*) precipitating salt and nitrate from a proportion of the caldillo produced by counter-current heat transference to refrigerated solution; (*b*) separating the clear liquor from the deposited crystals; and (*c*) producing a caldo by means of a further supply of caldillo from the leaching vat, in contact with the deposited crystals and the application of heat.

Cold nitrate brine, as a suitable medium for heat absorption from the first portion of the caldillo, would be available from an agua vieja refrigeration plant. With increasing temperature there would be no danger of crystallisation in the cold brine pipes. Heat transference would be arranged in a precipitation vat provided with stirrer arms, the agitation of the solution assisting heat transfer and preventing the deposition of crystals on any part of the apparatus. The separation of crystals from solution can be made in a grainer with mechanical rakes, as used in the salt industry.

Vacuum - pan Evaporation.—A considerable amount of work has been done in connection with the concentration of nitrate liquors in vacuum-pan evaporators, and reliable data are available which indicate the practicability of the method.

High-grade caldo, which is desirable under all circumstances, can only be produced by a corresponding high temperature. This indicates a difficulty in operating multiple-effect pans: the final liquor is dense and only circulated with difficulty, and there is a danger of the precipitation of nitrate in the high-vacuum effects. The problem of handling the large amount of salt deposited and the need for the frequent cleaning of the apparatus indicates the need for efficient control.

The success of vacuum-pan evaporation of water in the salt industry is largely the result of the utilisation of exhaust steam. The cost of producing power on the nitrate pampa is so high and the amount of exhaust steam available is so small that little advantage results by the installation of vacuum-pan evaporators. It would appear that the logical location for such apparatus would be at the coast. Considerable economies could be effected by the utilisation of exhaust steam from plants supplying current for mining or nitrate enterprises inland, local power requirements, or from the electrification of railways.

The solution to be evaporated at the port works would preferably be a refrigerated or cooled agua vieja, transported to the coast in pipe line or by means of oil tank cars, which are now returned to

the coast empty. The products at the port works would be commercial nitrate and water.

THE ADVANTAGES OF SUCH A SCHEME ARE:

1. Economical distillation of water, available for sale at the coast.
2. Saving in freight on fuel from port to pampa; and freight of a considerable proportion of total nitrate produced, from pampa to port.
3. Improved extraction of nitrate in the leaching plant by the avoidance of return of nitrate solution (agua vieja or agua feble); and more rapid treatment.
4. Concentration of power plant and evaporator work at the coast, where conditions are more favourable for keeping the high-class mechanical labour required, and for maintaining the equipment in satisfactory condition.
5. Major plant expenditure will be in connection with port works, which will be available for the treatment of brine from the nitrate fields in the vicinity, or between the port works and the nitrate plant.
6. Opportunity to purchase agua vieja from small oficinas near the railroad or pipe line at low rates, for all operators know that, by avoiding the return of agua vieja to the plant, greatly improved results can be secured.

THE EVAPORATION OF WATER FROM NITRATE SOLUTIONS.

The evaporation of water from nitrate solutions constitutes one of the major expenses of caliche treatment at the present time. The efficiency of any alternative to the Shanks process will depend largely on how cheaply the commercial product can be obtained from the solutions resulting.

Solar Evaporation.—Primary consideration must be paid to the possibility of utilising the extraordinarily favourable atmospheric conditions prevailing at all seasons of the year on the Chilean nitrate pampa—the heating effect of the sun, the abnormal dryness of the atmosphere, the persistence of high winds, and the almost entire absence of rainfall. These advantages point to conditions which are highly favourable to solar evaporation. The feasibility of utilising the evaporative powers of the air in conjunction with the sun-heating of thin layers of liquor at a negligible expense offers advantages that cannot well be disregarded. The problem confronting the industry as soon as the present high-grade deposits are nearing exhaustion will be in connection with the recovery of a commercial nitrate from a comparatively low-grade brine. The fractional crystallisation of such a solution by solar evaporation and the production

of an impure crystal must be inevitably an important phase of any logical scheme suggested.

The principal objection to the adoption of a system of solar evaporation in Chile is the porous, absorptive nature of the ground on the pampa when considered for such a purpose. The flats must necessarily cover a large area. If constructed of steel, the expense would be prohibitive. Concrete is generally unsuitable for nitrate solutions, especially in view of the fact that minor earthquake shocks are of frequent occurrence.

In practically all parts of the nitrate pampa a finely-divided clayey material is encountered, locally known as borra. It is proposed (patent applied for) to enclose suitable areas of flat or levelled ground with walls of masonry, borra pug, or other impervious mixture, at a small distance above and below the ground. Chuca, costra, caliche, or other earthy material in the vicinity, or obtained from the levelling of the ground, and containing the borra or clay mentioned, will be delivered to a type of mixing machine, and a pulp obtained by the addition of water. If the mixture contains over a certain percentage of sand or coarse material, the pulp will be classified—the coarser proportion will be removed by hydraulic or cone classifiers or by mechanical means, or by hand labour. The remaining liquid, containing the borra, will if necessary be thickened, and the excess of water thereby removed. Borra pulp obtained either in the manner described, or resulting from the treatment of caliche by the Shanks or other process

which results in a classification, will be pumped or delivered by other suitable means to the enclosure, over which it will be allowed to flow. As soon as a complete layer has been formed, of suitable thickness, the borra may be left exposed and so partly dried, so that it can be rolled and levelled. A top layer of sand, gravel, or other material may then be added for a surface; or the flat may be flooded with brine and the crystals resulting from subsequent evaporation may be utilised to form a protective bed.

Experiments have demonstrated that a considerable amount of commercial nitrate can be obtained by the solar evaporation of water from agua vieja, especially if the evaporated water is replaced at the same time by more agua vieja. Beyond a certain point an impure nitrate-salt crystal is produced, and at a later stage it would be profitable to return the brine liquor to the treatment plant.

Certain flats would be used for the crystallisation of a commercial product, the solution being pumped to other flats as soon as the percentage of salt being deposited rose above the economic limit. The commercial crystals would be centrifuged in the usual way, to lower salt percentage by the reduction of the amount of entrained brine in contact with the product; and then sacked or otherwise prepared for shipment. The impure crystals from the second series of flats would be delivered to the refining plant, where any available solution would be used to prepare a caldo by ad-

mixture with the impure crystals and the application of heat, the excess salt crystallising out as the density of the liquor increased, or remaining as an insoluble. The usual methods can be followed for the separation of the salt and the crystallisation of commercial nitrate from the caldo produced.

The harvesting of the crystals produced in the solar evaporation flats would be carried out by mechanical means, and by the use of apparatus the efficiency of which has been amply demonstrated in the salt industry. The most suitable machine for the purpose would loosen the deposit by means of a plough, elevate the crystals by chain and bucket elevator, and deposit them into cars.

Spraying of Caliche Piles.—Piles of caliche are exposed, at several stages in the handling of the material, to the hot sun and dry air of the pampa. It is suggested that advantage be taken of the absorptive nature of the atmosphere to accomplish some of the necessary evaporation by spraying the piles with agua vieja or other nitrate liquor. The disadvantage in the proposal is seen in the complication introduced in connection with the accounting of nitrate recovery.

It is not suggested that the system should form a fundamental phase of any scheme of treatment, or be relied upon for the evaporation of large amounts of water. On the other hand, the cost of the operation would be negligible.

Spray Evaporation of Water in Hot Nitrate Solutions, in Presence of Flue Gases, and above Moving Caliche Bed.—With an ability to regulate

the operation, an efficiency of about 75 per cent. of heat in flue gases may be expected if spraying is practised. If a considerable amount of water is required to be evaporated by this method, then the heat from the flue gases may be supplemented by oil burners. The solutions cannot be concentrated, but a system of operation to insure evaporation of water and the automatic handling of crystals produced might be arranged on the following lines:

The caliche to be fed to a storage bin after having been fine-crushed to the required size, and distributed over the width of a pan conveyor forming the floor of the spray compartment. This conveyor would deliver to a second conveyor system feeding the leaching vats.

The spray house, of which the pan conveyor forms the floor, would be provided with intake fans and free exit, or free intake and exit fans. The air passing into the spray house would be under strict control, with low velocity to prevent drift losses. The walls of the spray house would be thoroughly insulated to prevent heat losses. With the walls at a lower temperature than the solution it is improbable that any considerable deposition of crystals would occur there. Hot agua vieja could be used, being atomised by any of the various methods of spraying in use. Deposited crystals would fall on the moving caliche bed, to be mixed with the charge. It might be found desirable to regulate the agua vieja flow so that an evaporation of about 50 per cent. of the water content of

the solution would be obtained, and the deposited material would then be mostly salt.

A possible advantage of the scheme would be that operation could be made to produce a calcination of the borra particles in the charge. If a counter-current flow of flue gases and caliche was arranged in some type of tumbler, the borra particles would be released from the charge, exposed to maximum heat, carried forward to the spray chamber, where they would be trapped by means of the semi-evaporated brine spray possibly rendered less refractory for leaching treatment, and mixed with the charge in the identical proportions in which they originally existed.

Spray Evaporation of Water from Atmospheric-Temperature Solutions by means of Nozzles.— In such a system there can be no appreciable heat losses. But heat loss is a necessary concomitant to evaporation, so that with atmospheric-temperature solution and cold air it is obvious that evaporation will be insignificant in proportion to size of plant and power required. It has been found in practice that efficient control involves considerable supervision. A minute, impure, slime crystal is formed, which must be handled by means of settling and pumping equipment. Nozzle-spray evaporation under such conditions is only feasible with agua vieja or lower-grade solutions. The latter, however, are invaluable for dissolving and replacing purposes in leaching treatment, and their abstraction at any earlier stage of the process would interfere seriously

with the efficient extraction of nitrate from the caliche.

Appreciable evaporation (up to 5 per cent. of the water contained, per passage through the nozzle) can only be secured by an exceedingly fine atomisation of the solution, involving comparatively high pressures. Complete evaporation of water in individual particles of solution, therefore, results, and a considerable amount of salt is deposited. Chokage of the nozzles is of frequent occurrence with a solution of the density and other characteristics of cold agua vieja. The clearing of the pipe lines with steam tends to free iodine, and corrosion results, the scale produced causing continual trouble.

Evaporation in Multiple-Effect Vacuum Pans.—Full-scale evaporator plants are in operation on the Chilean pampa, and sufficient data are available for commercial plant design. Multiple-effect evaporators combine the advantages of caldo production, heat economy, water conservation, and quick evaporation in one step. By the use of caldillo—the first solution from a leaching plant—a solution is taken which has no value for replacement or dissolving purposes.

The disadvantages of vacuum-pan evaporators are seen in high first costs, possible danger through depreciation from iodine corrosion, and the frequent need for cleaning, thus reducing the evaporating efficiency and the capacity of the plant.

USE OF THE GRAINER FOR EVAPORATION AND CRYSTALLISATION PURPOSES.

Grainers with mechanical raking devices (see Fig. 2, patent applied for by inventor—G. B. Willcox) have been used extensively in the salt industry, and have recently been adopted for the cooling and evaporation of water from concentrated liquors made from the crystals produced by the fractional crystallisation (by solar evaporation) of potash brine. The size of the grainers now used is standardised, being 150 feet long, 12 feet wide, and 2 feet deep. Steel is generally used for the tray, and is recommended in place of concrete, especially in the case where hot liquors are being handled.

The raker is of light angle-iron construction with cross braces, at intervals, which carry scraper blades. A slow, long stroke is given to the mechanism by means of direct connection with the piston of an hydraulic cylinder. On the forward stroke the blades push the salt forward and up the incline. On the return stroke the blades feather the deposit. The brine forms the lubricant. Wearing plates on the side angles move on cast-iron shoes placed in the bottom of the grainer. The stuffing-box connection shown in the accompanying illustration is seldom used. The cylinder piston-rod extension

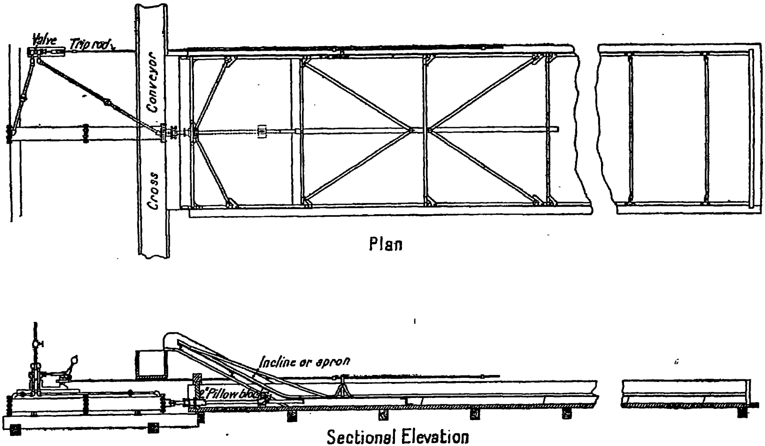

Fig. 2.—Plan and Sectional Elevation of Automatic Crystal Raker, with Hydraulic Drive.

[By courtesy of Willcox Eng. Co., Saginaw, Mich.

is usually connected direct to a light angle-iron structure, which is riveted to the tail end of the mechanism away from the salt discharge. Apart from this connection, the raker is entirely submerged.

Water pressure is used to operate the cylinder, the liquid being in continuous circuit through the cylinder and back to the pressure pump. Adjustment of stroke frequency or speed is made by opening or closing the valve leading to the cylinder.

The grainers operate in buildings in which quantity of air supplied is under control, and where minimum loss of heat occurs. At one potash refinery, three standard rakers, equipped with Willcox mechanisms, are used in series. The hot potash caldo enters at the head of the first grainer, near the crystal discharge, passes out through the tail of the first grainer into the tail of the second, out of the head of the second into the head of the third, and finally out of the tail of the third. During passage through the grainers the potash brine drops to atmospheric temperature. The collection of crystals is entirely automatic, the product is comparatively coarse, and carries about 8 per cent. moisture.

It would appear that, in the absence of any sudden chilling of the solution, all crystallisation begins on the surface of the liquor. Crystals formed in this manner are free and fall to the bottom. With ordinary grainer operation there appears to be no growth of crystals except at the sides, and if these were properly insulated it is suggested that no crystallisation would occur there.

No work appears to have been done in Chile with regard to the automatic handling of crystals deposited from nitrate solutions, in spite of the high cost of operation and clumsiness of the present methods. It is suggested that there is a field for the use of grainers,

FITTED ENTIRELY OR FOR A PORTION OF LENGTH WITH STEAM PIPES.

(*a*) In connection with the desalting and concentration of caldillo or other liquor.

(*b*) In connection with the final concentration and desalting, or desalting only, of vacuum-pan liquor.

WITHOUT STEAM PIPES.

(*a*) For evaporation and crystal deposition with continuous flow of solution.

(*b*) In connection with the evaporation of water from caldo, the utilisation of the majority of the contained heat, and without the deposition of a prohibitive amount of salt.

The advantages to be gained by the successful adoption of such a system would include :

1. Reduction in area of plant required.
2. Ability to grade product, from high-grade crystals containing caldo (with low salt percentage) as moisture, to the ordinary commercial nitrate.

3. Elimination of all hand labour, and the continuous and automatic delivery of crystals of varying grades, from pure nitrate to a commercial product.
4. Elimination of launder delivery of caldo for distances, with reduction of radiation losses, prevention of formation of small crystals and high moisture percentage in product.
5. Ability to control heat losses during earlier stage of cooling, so as to obtain maximum evaporation of water; and ability to hasten later stage when evaporation is slight.

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